

REPORT DOCUMENTATION PAGE					<i>Form Approved</i> <i>OMB No. 0704-0188</i>	
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1. REPORT DATE (DD-MM-YYYY) 19-04-2010		2. REPORT TYPE Final			3. DATES COVERED (From - To) 1 April 2007 - 30 November 2009	
4. TITLE AND SUBTITLE Diamond Nanowire for UV Detection					5a. CONTRACT NUMBER	
					5b. GRANT NUMBER FA9550-07-1-0140	
					5c. PROGRAM ELEMENT NUMBER	
					5d. PROJECT NUMBER	
6. AUTHOR(S) Professor J. Xu PhD Kim, Jin Ho PhD Hsu, Chih-Hsun Park, Hongsik Palefsky, Steven					5e. TASK NUMBER	
					5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Brown University, Division of Engineering, Box D, Providence, RI 02912					8. PERFORMING ORGANIZATION REPORT NUMBER 521008F	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) AF Office of Scientific Research 875 North Randolph Street. Room 3112 Arlington, VA 22203-1995					10. SPONSOR/MONITOR'S ACRONYM(S)	
					11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for Public Release, distribution is unlimited						
13. SUPPLEMENTARY NOTES						
14. ABSTRACT A number of important discoveries about diamond nanowires were made. First is the proof that this novel morphology of carbon can exist, and can be fabricated in a CVD process at atmospheric pressure. Raman spectroscopy, TEM, SEM, electron diffraction, and photoluminescence spectroscopy were used to characterize the NWs. These measurements showed that the nanowires are formed of diamond with high crystallinity and high uniformity, encased in a graphitic shell. DNWs were also fabricated using a RIE process. This method allows for reliable control of the shape, length, diameter, density, and doping of the DNW. Several other productive projects have arisen as offshoots of this work, including a theoretical study of the size- and material-dependence of electrical properties of metal-nanowire interfaces, an investigation of the memory effect of a representative NW structure (SWCNT on nitride-oxide), the fabrication and study of Bi NWs and Bi2S3 nanostructures, and the first discovery of Cooper pairs in an insulating phase of material: named one of the "ten top physics stories of 2007" by the American Institute of Physics.						
15. SUBJECT TERMS Diamond, nanowire, ultraviolet, detector, nanotechnology, carbon, optics, nano-electronics, transistor, field-emission						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UU	18. NUMBER OF PAGES 9	19a. NAME OF RESPONSIBLE PERSON J. Xu	
a. REPORT U	b. ABSTRACT U	c. THIS PAGE U			19b. TELEPHONE NUMBER (Include area code) 401-863-1425	

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1. Principal Investigator Name:

Jimmy Xu

2. Grant/Contract Title:

DIAMOND NANOWIRE FOR UV DETECTION

3. Grant/Contract Number:

FA9550-07-1-0140

4. Reporting Period Start (MM/DD/YYYY):

04/01/2009

5. End (MM/DD/YYYY):

02/28/2010

6. Program Manager:

Dr. Donald Silversmith, yr. 1- yr.3, and Dr. Brian Thomas, yr. 3.

7. Distribution Statement (as on SF-298):

Unrestricted.

8. Annual Accomplishments (200 words maximum):

Crystalline diamond nanowires have been grown for the first time. And the growth condition was 900C and atmospheric pressure - extraordinary for diamond growth. Moreover, individual diamond nanowires have been successfully extracted from growth substrates, transported, and placed on to test substrates. Pronounced UV photoluminescence has been obtained in a single diamond nanowire. Electrical contacts to diamond nanowires have been made, forming the first ever *diamond nanowire electronic device*. As happens in unprecedented explorations, rapid advances often encounter unexpected obstacles. Such has been the case here: I-V characteristics revealed a parasitic conduction on the diamond nanowire surface. On one hand, this presents a problem to gate control in a field-effect transistor operation. On the other hand, it also could help reduce a difficult problem – high contact resistance, common to all nanowire devices. The challenge has been to get the latter and avoid the former, and one that will be met with an improved fabrication process and a post-fabrication treatment which are in development. A theoretical model of the size- and geometry-dependent metal-nanowire contacts has been developed, which led to

the finding that the contact resistance is inherently greater in a nanowire than in a planar device of the same thickness. An effort to interface the hard diamond nanowire with a soft metallic material has been made, and resulted in a surprising discovery – bismuth when nano-patterned could undergo a phase transition from metallic to insulating. This discovery was cited as one of “Top Ten Physics Stories of 2007” by the American Institute of Physics Bulletin in its year-end issue.

9. Archival Publications (published) during reporting period:

In preparation:

1. Paper on UV photoluminescence spectrum of CVD-grown diamond nanowires (target: Applied Physics Letters)
2. Paper on field emission of CVD-grown diamond nanowires (target: Advanced Materials)
3. Paper on "Functional Dependence of Electrical Contact Properties on the Geometry of Semiconductor Nanowires" (target: Physical Review B)

In review:

1. Chih-Hsun Hsu, Sylvain Cloutier, Steven Palefsky, Jimmy Xu. Synthesis of Diamond Nanowires using Atmospheric-Pressure Chemical Vapor Deposition. (In review, at Nano Letters.)

Published:

1. Jin H. Kim, Hongsik Park, Chih-Hsun Hsu, and Jimmy Xu, “Facile Synthesis of Bismuth Sulfide Nanostructures and Morphology Tuning by Biomolecule”, accepted to J. Phys. Chem., April 14, 2010
2. Hongsik Park, Hyunjung Shin, Jin Ho Kim, Seungbum Hong, and Jimmy Xu, "Memory effect of a single-walled carbon nanotube on nitride-oxide structure under various bias conditions," *Applied Physics Letters*, vol. 96, 023101, Jan 11 2010.
3. M. D. Stewart, Nick Yin, Jimmy Xu, Jim Valles, "Superconducting pair correlations in an amorphous insulating nanohoneycomb film," *Science*, vol. 318, pp. 1273-1275, Nov 23 2007. (This unexpected discovery was referred to as “finding superconducting Cooper pairs in insulator” by American

Institute of Physics in its year-end bulletin and cited as one of the “Top Ten Physics Stories of 2007”).)

10. Changes in research objectives (if any):

None

11. Change in AFOSR program manager, if any:

It was in the program managed by Dr. Donald Silversmith. In yr-3 it was transitioned to Dr. Brian Thomas.

12. Extensions granted or milestones slipped, if any:

The remarkable findings broke new ground for advancing basic science, and also opened opportunities and challenges that were not (or could not have been) expected. What opened up following the two extraordinary discoveries were the questions of mechanisms. Understanding the mechanisms is critical to further advances and applications, and essential to reproducibility and improvement in the case of the diamond nanowire growth. In this context, we decided to stay focused on the mission of identifying and understanding the interactions of the key parameters and processes, and not rushing into establishing claims of applications and devices. The latter is however important and will be pursued aggressively when a basic understanding of the extraordinary discoveries is developed which is anticipated in the next phase. As a result, a working diamond nanowire UV detector can be expected within the coming few months. And, a completely new diamond-bismuth heterojunction system for rectifying photocurrent response from UV to mid-IR is under development.

Final Technical Report on
DIAMOND NANOWIRE FOR UV DETECTION

FA9550-07-1-0140

To Dr. Donald Silversmith and Dr. Brian Thomas

AFOSR

PI: Jimmy Xu

Brown University

184 Hope St., Providence, RI 02912

Objectives:

To prove the existence of diamond nanowires, which if proven would represent yet another new phase of carbon after the discoveries of carbon nanotubes and carbon-60. To fabricate, extract, transport, nano-scale position, and contact individual diamond nanowires, and test and characterize their optical responses, and then to assess their potential for UV and optical applications.

Status of Effort:

The findings and results of this project are many, the most important of which is perhaps that of the CVD grown diamond nanowires. This constitutes the first definitive proof that there exists yet another new morphology of carbon. Also remarkable is the fact that it can be grown in a chemical vapor deposition (CVD) process at atmospheric pressure and 900C. Comparable to the well known ultra high pressure and ultra high temperature conditions of synthetic diamond growth, this new growth process is truly extraordinary, and is attributed to the nano-scale curvature of the effective growth chamber – a graphitic shell of 20 to 30nm radius. A number of experimental investigations were carried out to validate the diamond crystallinity of the grown nanowires, including Raman spectroscopy, transmission electron microscopy (TEM), scanning electron microscopy (SEM), selective area electron diffraction (SAED), electron energy loss spectroscopy (EELS) and photoluminescence spectroscopy. These measurements demonstrated that the nanowires are formed of diamond of high crystallinity and high uniformity, and that they are encased in a graphitic shell of 40nm to 50nm diameter.

For comparison and device concept exploration, diamond nanowires were also fabricated from bulk diamond films using a reactive ion etching (RIE) process. This method allows for lithographic control of the shape, length, diameter, and doping of the diamond nanowires, and therefore provide a platform on which diamond nanowire device applications can be explored in parallel with the further development the direct diamond nanowire synthesis process for controllability, yield, and reproducibility.

Several other productive projects have arisen responding to the new opportunities opened by the advances made in this exploration. These projects include a theoretical study of

the size- and geometry-dependence of the metal-nanowire interfaces, an investigation of the memory effect of a nanowire on oxide, aided by a model system of a single-walled carbon nanotube placed on nitride-oxide, the fabrication and study of bismuth nanowires and Bi_2S_3 nanostructures that are to form a heterojunction with the diamond and capable of stand-alone operation, and the first discovery of a phase dramatic transition from metallic to insulating in a nano-patterned bismuth. The last of which was cited as one of the “ten top physics stories of 2007” by the American Institute of Physics in its year-end bulletin.

Accomplishments/New Findings:

The discovery of diamond nanowires is important for a number of reasons. Among the discovered carbon allotropes, diamond has long been a material of interest for research due to its unique properties. Its wide band gap, high electron and hole mobility and negative electron affinity (NEA) make it an attractive candidate for use in ultraviolet (UV) light detectors and emitters[2]. Of all known materials, it is the hardest, and has the highest thermal conductivity. These properties also make diamond suitable for applications such as radiation particle detectors[3], field effect transistors[4], electron field-emission sources[5], position-sensitive biochemical substrates[6] and many other possible applications. Due to diamond's robustness, these technologies could be applied in harsh environments such as high temperatures or high-power devices for space applications.

Diamond nanowires have been a subject of great interest in various theoretical studies and structural simulations with findings providing insights into the structural stabilities and inspiration for potential applications [7-9]. Synthesis of crystalline diamond nanowires is of major interest since they offer the potential for enabling applications across many disciplines, for advancing the science of material synthesis at the nano and atomic scales, and for validating the search of new forms of carbon. However, the fabrication of long, single crystalline diamond nanowires using conventional thermal CVD methods has so far proven elusive, despite the potential benefits.

By altering an otherwise standard carbon nanotube (CNT) growth process with the introduction of sample cooling under a pure hydrogen flow, diamond nanowires were grown on a silicon substrate. They were straight, thin and long, and longitudinally uniform in exterior diameter (70-90 nm) along the entire lengths of tens of microns (Fig. 1).

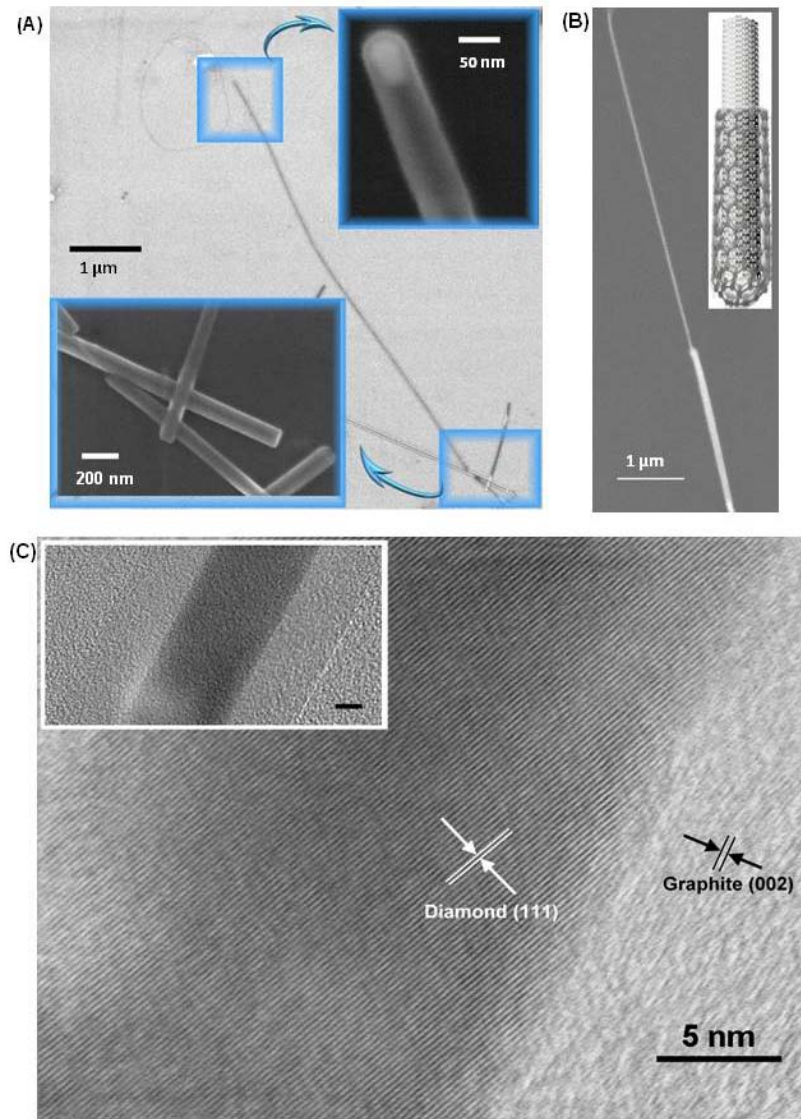


Fig. 1. Electron microscopy of diamond nanowires encased within a carbon nanotube shell. **(A)** Diamond core enclosed in a CNT sheath, typically tens of microns in length and 70-90 nm in exterior diameter. A magnified SEM image of the nanowire tip in the upper inset shows a catalyst embedded inside the tip of the nanowire. The SEM image in the lower inset shows the straight and uniform nanowires with flat terminations. **(B)** SEM image of a core-shell diamond nanowire with a portion of the CNT shell stripped away selectively by laser burning. The blurring of the diamond nanowire tip is due to oscillations caused by charge accumulation from the impinging electron beam. **(C)** The transmission electron microscopy (TEM) image shows the diamond core and CNT walls of the nanowire. The diamond (111) lattice (0.21 nm) is enclosed by the outer CNT shell with a graphite (002) surface (0.34 nm) whose surface stress provides a very high effective pressure[1] (on the order of GPa) in which the diamond phase is stable. The low-magnification TEM image in the inset shows the core-in-shell wire structure more clearly. The scale bar in the inset is 5 nm.

The growth process began with methane and hydrogen flow over an Fe catalyst solution dispersed on a silicon substrate under atmospheric CVD conditions at 900°C. After this process was completed, and without pumping the residual methane from the chamber, pure hydrogen was flowed through the quartz tube chamber at the rate of 200 sccm while the temperature was slowly lowered to ambient at the rate of ~1.2 °C/min over a period of 12 hours.

Various tests were performed to confirm the diamond structure of the nanowires and investigate their properties. Scanning electron microscope (SEM) imaging (Fig. 1A) shows sparsely-distributed straight nanowires that have a distinctively different look from the curly carbon nanotubes normally grown in CVD. The nanowires measure tens of microns in length and 70-90 nm in outer diameter.

The core-shell structure of a diamond nanowire encased in a graphitic shell manifested itself in an SEM image where a portion of the nanotube shell was stripped away by a focused 532 nm laser beam in the micro-Raman spectrometer as shown in Fig. 1B. Note that the diamond nanowire remained intact even after being subjected to prolonged laser exposure, indicative of the difference in light absorption between the transparent wide bandgap diamond and the narrow bandgap graphitic shell. This is also suggestive of a good crystallinity of the diamond nanowire.

High-resolution transmission electron microscopy (HRTEM) imaging of the nanowire was performed, as shown in Fig. 1C. It confirms that the structure of the nanowire indeed consists of a diamond core encased within a graphitic shell. From this image, we estimate an inner nanowire radius of ~15 nm and the thickness of the sheath to be ~20 nm, yielding the exterior diameter of the diamond nanowire to be 70 nm. In some cases, we observed amorphous carbon layers instead of a graphitic shell; however, the diamond nanowires contained within were of the same high-crystalline quality.

Information about the diamond structure of the nanowires was extracted from transmission electron microscopy. Figure 2A shows an HRTEM image of one of these nanowires, which reveals a crystalline diamond wire structure with a lattice constant of 0.21 nm, corresponding to the (111) orientation of diamond. This image indicates that

the size of the crystal domain is at least 30 x 30 nm, which is larger than predicted by theory [9]. In fact, in some cases, a single crystal with low defects was seen along the length of the entire nanowire.

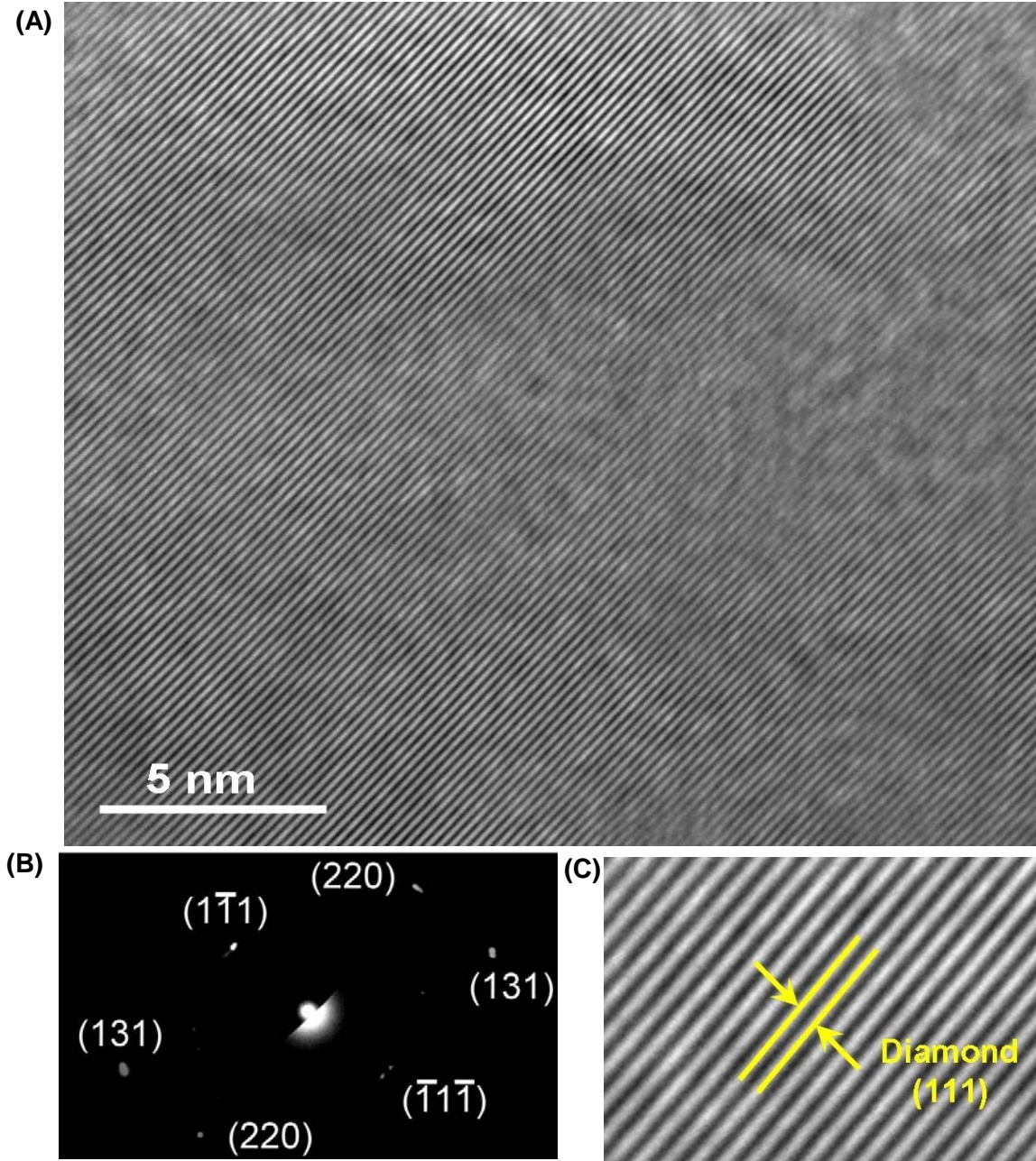


Fig. 2. High resolution transmission electron microscope (HRTEM) images of a single diamond nanowire. **(A)** HRTEM image showing the single-crystallinity of a diamond nanowire with domain size at least 30 x 30 nm in this micrograph. **(B)** The

selective area electron diffraction (SAED) pattern taken from the same diamond nanowire also shows a cubic polycrystalline diamond (c-diamond) structure. (C) A zoomed-in view of the crystalline structure displaying clear lattice planes of cubic diamond (111) surfaces with a lattice constant of 0.21 nm.

Figure 2B presents a selective area electron diffraction pattern (SAED) taken from the diamond nanowire along the $[-1,1,2]$ zone axis, showing that the nanowire has a crystalline cubic diamond structure. Electron diffraction patterns taken along multiple zone axes were further examined from the same sample, and again the cubic diamond structure was observed. The SAED experiment also indicated that diamond nanowire has the same lattice parameter as bulk cubic crystalline diamond. The diffraction pattern suggests that the growth direction of this nanowire is along the $[2,2,0]$ principal axis.

Micro-Raman spectra, taken with a 532 nm laser beam focused on randomly-selected micro-spots of individual isolated nanowires (e.g. at the point indicated by a red cross in the inset of Fig. 3), reveal the signature diamond peak[10] at 1332 cm^{-1} , dominant in intensity and extremely narrow in linewidth. Its FWHM of 14.5 cm^{-1} is comparable to that of high quality CVD-grown bulk diamond ($5\text{ to }10\text{ cm}^{-1}$) and is much narrower than that of other diamond nano-crystallite structures found in literature[11]. The two additional peaks at 1564 cm^{-1} and 1603 cm^{-1} are those of the well-known graphitic G-bands[12]. Since the micro-Raman spectroscopy was performed on individual isolated nanowires as shown in Fig. 3, these G-band peaks support the HRTEM evidence of a graphitic shell and a diamond core-in-nanotube shell structure. No such spectral lines were observed from the substrate when moving the excitation beam away from the wire. A Raman line at 1150 cm^{-1} often seen in CVD-grown nanodiamond films is absent here, as is its associated 1450 cm^{-1} peak. These lines, if present, are usually indicative of poor quality diamond crystals[11].

Electron energy loss spectroscopy (EELS) was used as another confirmation of the diamond structure of the nanowires. The low-loss EELS spectrum (Fig. 4A) contains a sharp peak which is known to be a product of the σ plasmon in diamond. The high-loss

spectrum (Fig. 4B) has features at approximately 285 and 295 eV, which come from K-shell electrons in diamond.

Photoluminescence spectroscopy was performed on the diamond nanowires. With the nanowires on a quartz substrate, the sample was illuminated with a 337 nm nitrogen laser. The resulting emission spectrum, shown in Fig. 5, shows a strong peak at 415 nm. This feature could indicate the presence of the N3 center in diamond[13]. This defect is modeled by three substitutional nitrogen atoms and a vacancy[14].

Using a nanomanipulator in an SEM, the field emission data for one isolated diamond nanowire was measured, and this is shown in Fig. 6 as an I-V plot, with an inset showing the almost-linear Fowler-Nordheim (F-N) relationship, and confirming the electron field emission behavior. To compare the field emission property qualitatively with reported data of a single carbon nanotube published, the field enhancement factor was estimated using the approach reported by Xu *et al.*[15]

From the measured F-N characteristic, it was found that the field enhancement factor was $\beta \sim 60000$ [16], which is almost two orders of magnitude larger than the factor from carbon nanotubes (~ 1000) reported by Xu[15].

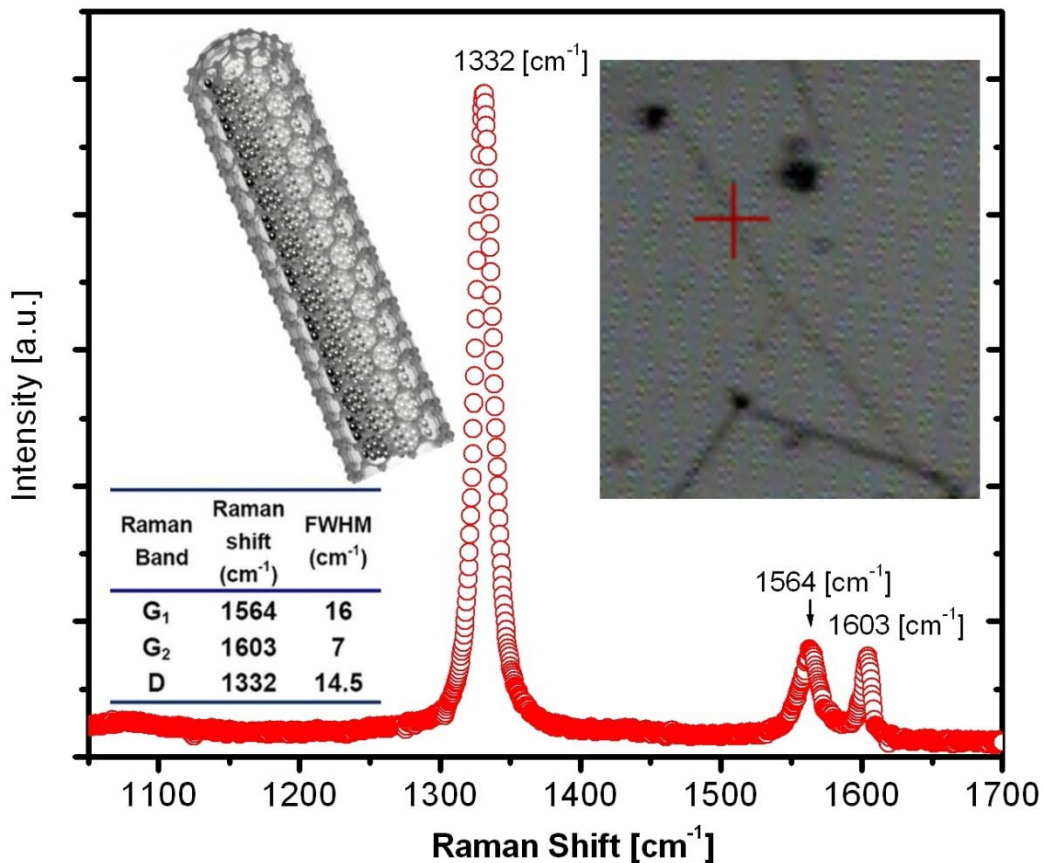


Fig. 3. Micro-Raman spectrum taken from the same nanowire shown in the SEM image (Fig. 1A). The diamond signature peak at 1332 cm^{-1} is narrow and pronounced, and the two Raman peaks at 1564 cm^{-1} and 1603 cm^{-1} demonstrate the presence of a CNT shell. The inset is the optical microscope image of the diamond nanowire placed in the Raman system corresponding to the SEM provided in Fig. 1A. The cross hair in the inset marks the location where the Raman laser beam (spot size $\sim 1\text{ }\mu\text{m}$) impinged on the diamond nanowire sample. This figure also includes (top left) a graphic impression showing formation of the nanowire within a shell.

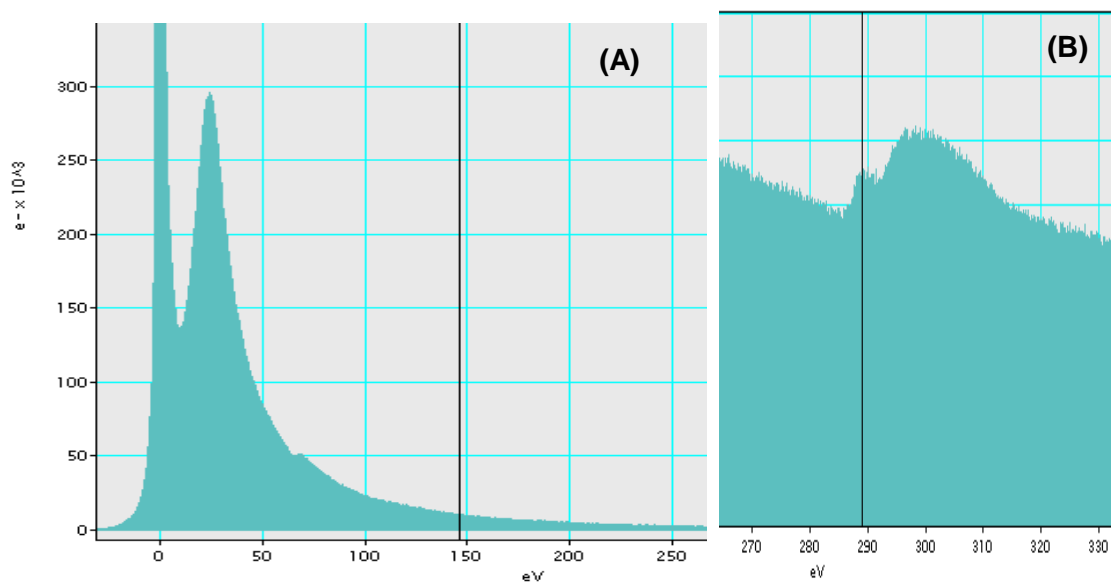


Fig. 4. Electron energy loss spectroscopy (EELS) of diamond nanowire. **(A)** Low-loss region. The peak at is the known signature of the σ plasmon in diamond. **(B)** High-loss region. The features at ~ 285 and $\sim 295\text{ eV}$ are indicative of the K-shell of diamond.

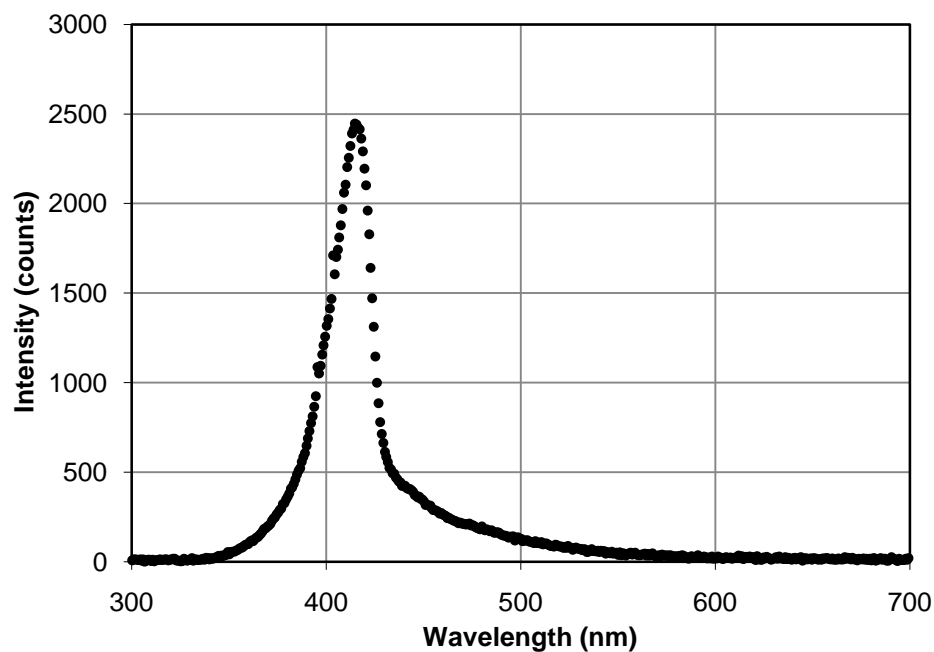


Fig. 5. Photoluminescence spectrum taken from diamond nanowires on quartz substrate, showing peak at 415 nm. Sample excited by 337 nm nitrogen laser. This feature suggests the presence of the N3 center in diamond, which can be modeled by three substitutional nitrogen atoms and a vacancy[14].

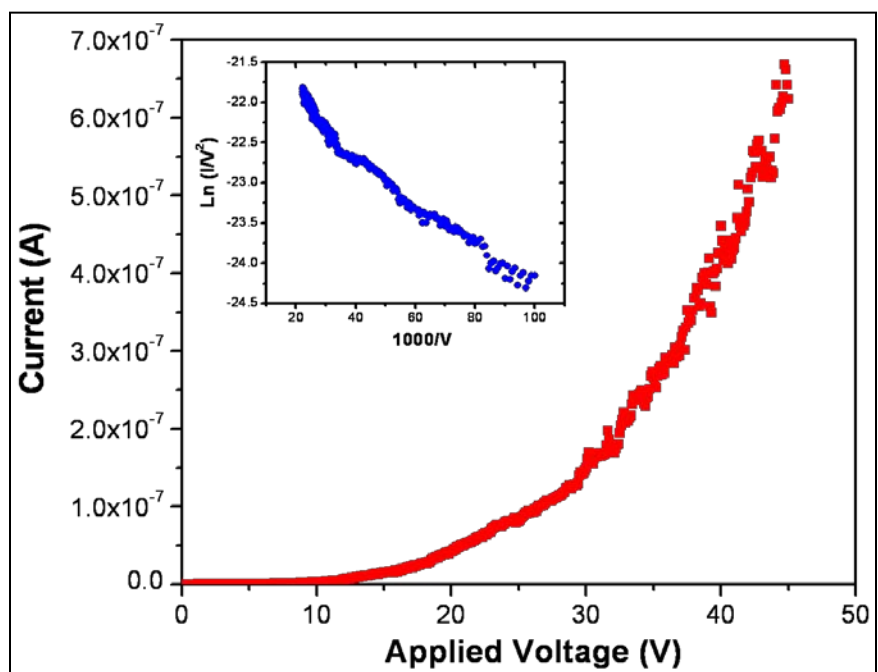


Fig. 6. Typical field emission data obtained from individual diamond nanowire in SEM by nanomanipulator. The inset presents the emission current data plotted in Fowler–Nordheim coordinates, demonstrating the field emission behaviour of the diamond nanowire.

There is an existing theoretical framework within which this discovery may be placed; stable diamond nanowires have been shown to be possible in computational models and simulations for some time (albeit under idealized theoretical settings and without considering the actual growth conditions and environmental limitations). More specifically, density functional models have shown that diamond nanowires might exist in cubic, cylindrical or dodecahedral crystalline forms[7]. It seems reasonable to expect that the nano-scale curvature has played a significant role, as it is known that a small diameter is accompanied by a large surface stress, which could give rise to a high equivalent pressure to the core of the nanowire. Indeed, an empirical thermodynamic model argued that it is possible for a diamond nanowire to grow inside a tubular graphitic enclosure (or carbon nanotube)[9]. A key aspect of this argument, independent of the specifics of the model, is that for a small-diameter wire a capillary pressure (or so-called Young-Laplace pressure) $\Delta P \approx 2\gamma/r$ (where r is the radius) can be sufficiently high to allow the diamond phase to exist in the inner core while the outer shell with less equivalent pressure takes on the graphitic phase. Depending on the specific surface and interface energy parameters, within a thick nanotube-like shell of an interior radius around 10-25 nm, or for even smaller dimensions occurring at a defective spot of the nanotube wall, the equivalent pressure could reach the order of 1~5 GPa[17]. Similar phenomena were observed experimentally in diamond nucleation inside carbon onion high curvature enclosures[18]. The use of carbon nanotubes as high-pressure cylinders that can cause large pressure (as high as 40 GPa) buildups within the nanotube cores which can plastically deform, extrude, and break solid materials that are encapsulated inside the core was reported as well[1].

Molecular dynamic (MD) simulation[8, 19] estimates that a stable diamond nanowire could have a diameter on the order of ~10 nm. It is probably not so meaningful to seek quantitative agreement between theory and experiment at this stage. For one thing, the boundary conditions set in the models are far simpler and fewer than the experiments[7],

and the interface conditions are yet to be determined. However, it is worth noting that the order of magnitude estimate does correspond well to the experimental findings of the wire diameter (Fig. 1C) and the crystal domain size (Fig. 2A).

As with the discovery of all new materials and systems, the technologies that will emerge from diamond nanowires cannot be anticipated at such an early stage. For example, carbon nanotubes are now pursued for many applications that were not anticipated at the time of their discovery, or predicted for years after. So was the case for lasers. However, one may already see a number of potential applications of this new material, in addition to the stated goal of solar-blind, radiation-hard diamond nanowire UV detectors[3].

The use of diamond nanowires in field-effect transistors could be advantageous for high-power, high-temperature, and high-speeds[20] due to their one-dimensional structure, high mobility, large band gap and breakdown field, and high thermal conductivity[21]. DNA sensors[22] would also be possibilities of broad interest. Finally, diamond nanowire has been computationally simulated to possess a brittle fracture force and a zero strain stiffness that exceeds carbon nanotubes for radii greater than about 1-3 nm[18].

To aid the exploration of the growth conditions, a new fully-customized CVD furnace (Fig. 7) was designed in-house, assembled externally by a contractor, and installed by ourselves. This system allows for a much better-controlled environment than the previous system, and provides many features that were not available in the old system, such as: the ability to dope samples, liquid and metal-organic injection, RF plasma, and field-guided growth. The new furnace will make the search for reliable, controllable, high-yield growth conditions a simpler task.

There are several projects that have branched off from the diamond nanowire initiative. A study of nanopatterned bismuth resulted in the discovery that Cooper pairs can exist in material in an insulating state, whereas it was previously thought they could exist only in a superconductor[23]. This finding was named one of the ten top physics stories of 2007 by the American Institute of Physics. A theoretical study has been performed to model the size- and geometry-dependence of electrical properties of metal-NW interfaces. One

finding is that the contact contribution to resistance is much more dominant in some cases than was previously thought.

An investigation of the memory effect of a single-walled carbon nanotube (SWNT) on nitride-oxide structure (NOS) under various bias conditions showed that a SWNT-NOS structure could potentially be used in nonvolatile memory cells[24]. Other investigations of bandgap-engineered nanowires include fabrication and study of bismuth nanowires, whose bandgap varies with diameter, and the bio-molecule-aided growth of Bi_2S_3 nanostructures, which can have totally different morphologies depending on the sulfur source, and have demonstrated field effect modulation.

Since the discovery of CVD-grown diamond nanowires, a great effort has been made in order to best showcase the potential of the new material system developed in this work. When the first manuscript was returned from reviewers at Nature, their positive response indicated a great interest in the findings, but also a request for more and better evidence. Efforts to make the best possible case intensified, and the measurements and images described above were the product of countless man-hours of work. With each successive submission to Nature, the paper was improved, and reviewers expressed little doubt about the importance of the work, but the review process took very long. In the end, we decided to simply submit the key findings to Nano Letters, which is currently in review. This discovery is of great importance from both a fundamental science standpoint and an applications standpoint, and the use of diamond nanowires for UV detection should be an important technological breakthrough.

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Personnel Supported:

Jimmy Xu, principal investigator; Chih-Hsun Hsu, Hongsik Park, Steven Palefsky, graduate students; Dr. Jin Ho Kim, Postdoctoral Research Associate.

Publications:

In preparation:

1. Paper on UV photoluminescence spectrum of CVD-grown diamond nanowires (for Applied Physics Letters)
2. Paper on field emission of CVD-grown diamond nanowires (for Advanced Materials)
3. Paper on "Functional Dependence of Electrical Contact Properties on the Geometry of Semiconductor Nanowires" (for Physical Review B)

Submitted:

1. Chih-Hsun Hsu, Sylvain Cloutier, Steven Palefsky, Jimmy Xu. Synthesis of Diamond Nanowires using Atmospheric-Pressure Chemical Vapor Deposition (under review in Nano Letters)

Published:

1. Jin H. Kim, Hongsik Park, Chih-Hsun Hsu, and Jimmy Xu, "Facile Synthesis of Bismuth Sulfide Nanostructures and Morphology Tuning by Biomolecule", accepted to J. Phys. Chem., April 14, 2010
2. Hongsik Park, Hyunjung Shin, Jin Ho Kim, Seungbum Hong, and Jimmy Xu, "Memory effect of a single-walled carbon nanotube on nitride-oxide structure under various bias conditions," *Applied Physics Letters*, vol. 96, 023101, Jan 11 2010.
3. M. D. Stewart, Nick Yin, Jimmy Xu, Jim Valles, "Superconducting pair correlations in an amorphous insulating nanohoneycomb film," *Science*, vol. 318, pp. 1273-1275, Nov 23 2007. (This unexpected discovery was referred to as 'finding superconducting Cooper pairs in insulator' by American Institute of Physics in its year-end bulletin and cited as one of the "Top Ten Physics Stories of 2007".)

Interactions/Transitions:

Magnolia Optics Ltd, Dr. Ashok Sood and his team.

RPI, Professor Michael Shur and his team.

SUNY-SB, Professor Serge Luryi, and his team.

FSU, Professor Harry Kroto, (known for the Nobel prize discovery of C₆₀), had the first look of the findings as they were coming out from our experiments.

International collaboration, with Dr. Ki-Bum Kim of Seoul National University (AIPEL lithography of diamond films).

Awards/Honours:

Superconducting Pair Correlations in an Amorphous Insulating Nanohoneycomb Film –
Cited in the 2007 year-end Bulletin of American Institute of Physics (AIP) as one of the
‘Ten top physics stories of 2007’

Jimmy Xu, PI, elected AAAS Fellow, IEEE Fellow, appointed as World-Class University
Professor of Seoul National University (where he served a sabbatical)